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Title of Invention: METHOD FOR GRANULATING CARBOXYMETHYLCELLULOSE ETHER ALKALI SALT AND GRANULAR CARBOXYMETHYLCELLULOSE ETHER ALKALI SALT

[0022]

Namely, after the ether reaction, the CMC is maintained in a fibrous state during crude CMC production, and is a low density having bulkiness., Normally, desalinization and purification is performed with a low content aqueous solvent for the purpose of removing impurities such as salt byproduct contained in this crude CMC. However, with the present invention, as previously stated, the moisture content is desalinized and purified with a 40 to 70% highly aqueous IPA before being provided for granularization in a liquid. If this highly aqueous IPA which performs desalinization processing is filtered and separated, wet clumps of purified CMC containing 45 to 65% moisture can be obtained. By this process, the fibrous CMC containing moisture, adheres and is changed from soft jelly-like to hard jelly-like clumps of a solid substance. This solid substance is not dissolved by a IPA-based solvent, and, can be maintained in a state having good dispersibility by the stirring.

[0023]

Next, after filtering and separating, the hard jelly-like clumps of purified CMC are injected with 20% or less of an aqueous IPA (solvent) for the purpose of granularization in a liquid and the dehydration of clumps of purified CMC, wherein the purified CMC is stirred and mixed by rotating a stirring blade. Therefore, the clumps of purified CMC can be immediately granulated in a liquid, and can be granularized and cleaved to a particle diameter in the range of about 1 mm. At the same time as granulation, dehydration can be promoted by moisture migration to the IPA and a spherical granule can be obtained by rotating.

[0024]

The granular CMC obtained thereby has 80% or more of the particles in a particle size in the range of a particle diameter of 149 to 2000 μm, without containing a fine powder of a particle diameter 75 μm or less. Or, even if it contains a fine powder of a particle diameter 75 μm or less, it is a remarkably small amount in the range which does not bring about a reduction of the solubility by the adverse effects of dust and undissolved lump production. The measurement of the aforementioned particle diameter was measured by a standard sieve (described in JIS Z8801), and the entire particle size distribution could be verified. The granular CMC constructed with this kind of particle size distribution had a bulk density of 0.4 g/ml or more, specifically, a high bulk density of 0.4 to 0.8 g/ml.

[0033]

[Examples 1 to 15 and Comparative examples 1 to 6]

Using the purified CMCs 1, 2, and 3 created as mentioned above, granularization by the present invention was performed as follows. Namely, first, as shown in FIG. 1, a large sized mixer type mixer (manufactured by Kokusan Co. Ltd.) of a 10 liter volume equipped with a knife-like stirring blade 1 was used, and one part of solid content of the clumps of purified CMC and eight times the volume of 10% aqueous IPA shown in Table 1 below were added thereto. The content ratios of the CMC, IPA, and moisture of the entire system at this time are shown in Table 1 below. Immediately, stirring was begun at room temperature (25°C), and stirring was performed by setting the Reynolds number indicating the flow of the solution to the value shown in Table 2 below. The granularization conditions by the aforementioned mixer are shown in Table 2 below. Note that, the aforementioned Reynolds number is the value calculated by the Formula (1), and each value of the factors, blade diameter D (cm), blade circumferential speed U (cm/sec), liquid density ρ (g/cm^3), and liquid viscosity μ (g/cm·sec) which are necessary for the calculation of the Reynolds number are shown together in Table 2 below.

[0034]

[Table 1]

		Purified CMC type	Content ratios of the entire system (%)		
			CMC	Moisture	IPA
Ex.	1	2	7.84	27.51	64.65
	2	2	7.84	27.51	64.65
	3	2	7.84	27.51	64.65
	4	2	7.84	27.51	64.65
	5	3	8.45	22.97	68.58
	6	3	8.45	22.97	68.58
	7	3	8.45	22.97	68.58
	8	3	8.45	22.97	68.58
	9	1	8.83	21.18	69.99
	10	1	8.83	21.18	69.99
	11	1	8.83	21.18	69.99
	12	1	8.83	21.18	69.99
	13	1	8.83	21.18	69.99
	14	3	8.45	22.97	68.58
	15	2	7.84	27.51	64.65
Comp. ex.	1	2	7.84	27.51	64.65
	2	3	8.45	22.97	68.58
	3	1	8.83	21.18	69.99
	4	2	7.84	27.51	64.65
	5	3	8.45	22.97	68.58
	6	1	8.83	21.18	69.99

[0035]

[Table 2]

		Purified CMC type	Granularization conditions depending on mixer type						
			D	U	ρ	μ	Reynolds number	Stirring time	Solution temp
Ex.	1	2	10	31.9	0.94	0.25	1200	10 min.	20°C
	2	2	10	106.4	0.94	0.25	4000	10 min.	20°C
	3	2	10	266.0	0.94	0.25	10000	5 min.	20°C
	4	2	10	797.9	0.94	0.25	30000	5 min.	20°C
	5	3	10	37.5	0.96	0.30	1200	10 min.	30°C
	6	3	10	125.0	0.96	0.30	4000	10 min.	30°C
	7	3	10	312.5	0.96	0.30	10000	5 min.	30°C
	8	3	10	937.5	0.96	0.30	30000	5 min.	30°C
	9	1	10	48.5	0.99	0.40	1200	10 min.	20°C
	10	1	10	161.6	0.99	0.40	4000	10 min.	20°C
	11	1	10	404.0	0.99	0.40	10000	5 min.	20°C
	12	1	10	1212.1	0.99	0.40	30000	5 min.	20°C
	13	1	10	40.4	0.99	0.40	1000	15 min.	25°C
	14	3	10	31.3	0.96	0.30	1000	15 min.	25°C
	15	2	10	26.6	0.94	0.25	1000	15 min.	25°C
Comp. ex.	1	2	10	16.0	0.94	0.25	600	10 min.	20°C
	2	3	10	18.8	0.96	0.30	600	10 min	20°C
	3	1	10	24.2	0.99	0.40	600	10 min	30°C
	4	2	10	25.3	0.94	0.25	950	15 min	30°C
	5	3	10	29.7	0.96	0.30	950	15 min	30°C
	6	1	10	38.4	0.99	0.40	950	15 min	30°C

[0036]

Regarding the effect of the aforementioned stirring, the clumps of purified CMC were rotated in the liquid while being finely granulated in an aqueous IPA liquid, thus, a granular CMC which formed small spherical granules could be obtained. Note that, the optical photomicrograph of the granular CMC of the product of Example 1is shown in FIG. 2. It is understood that the granular CMCs obtained by granularization in a liquid in this way are all approximately spherical. Further, the small amount of moisture contained in the clumps of purified CMC moved into

the IPA liquid during the aforementioned stirring. As a result, the granularized CMC surface was dehydrated and keratinized, and an increase of the solidification and density occurred. Because of this, the CMC granule is difficult to attach to the devices in loose state, and a satisfactory CMC granularization product could be obtained. Therefore, the aforementioned granularization processing can be performed for 5 to 20 minutes, and, can be performed by either the continuous type or the batch type method.

[0037] After the aforementioned granularization processing and after dehydrating and separating by a centrifuge, drying was performed with hot air at 100°C for two hours with a warm air dryer. A granular CMC was obtained thereby.